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Feasibility of Developing Low-Power, Low-Current Methane Sensors for the Intrinsically Safe Mine Monitoring System

J. R. Stetter, W. R. Penrose, S. Zaromb, and A. F. Cohen

prepared for U. S. Bureau of Mines



ARGONNE NATIONAL LABORATORY

Energy and Environmental Systems Division

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FEASIBILITY OF DEVELOPING
LOW-POWER, LOW-CURRENT METHANE SENSORS
FOR THE INTRINSICALLY SAFE MINE MONITORING SYSTEM

by

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Energy and Environmental Systems Division

Geochemistry Section

June 1983

work sponsored by U.S. BUREAU OF MINES Pittsburgh Research Center

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EXECUTIVE SUMMARY

The commercial, scientific, patent, and technical literature has been surveyed to determine (1) if any existing methane sensors satisfy the Bureau of Mines (BOM) criteria for the Intrinsically Safe Mine Monitoring System (ISMMS) and (2) if such sensors do not exist, if it is feasible to produce them given present technology.

The ISMMS requirements for very low current and low power consumption eliminated some sensors and sensing modes that otherwise satisfied the criteria. Reconfiguration of some existing sensors could bring them within the ISMMS limits for power consumption (maximum of 16 mA at 8-18 V dc); however, the literature revealed no attempts by industry to reduce power dissipation to such low levels. Recent laboratory studies at Argonne National Laboratory and elsewhere suggest that developments in electrochemical technology could provide a basis for the design of very-low-current electrochemical sensors for methane.

The two major conclusions of this study are that (1) no device now available, in either the commercial or the development stage, can meet the ISMMS criteria and (2) given the present state of knowledge, devices that can meet ISMMS specifications probably can be developed in the near future.

The choice of the several possible approaches to sensor development should be based on analysis of risks and benefits. The three types of approaches recommended for further investigation are:

- Direct Electrochemical Detection. Sensors employing this detection mode, although not commercially available, are considered feasible, based on literature data and ANL research. The potential of such sensors can be investigated quickly and inexpensively.
- 2. Reconfiguration of Present Hot-Wire Sensors. There is no evidence that industry has attempted to minimize the current consumption of present hot-wire devices. This report discusses several approaches to sensor design that could significantly reduce current consumption; however, studies will be needed to determine if these changes will compromise sensing capabilities.
- 3. Combined Electrochemical and Solid-State Sensors. The most certain approach for development of a methane sensor that meets the ISMMS criteria is based on electrochemical sensing of partial oxidation products; the feasibility of this approach has been demonstrated. Measures will be needed to reduce power consumption by the catalyst, but this reduction appears to be possible.



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ABSTRACT

Low-power, low-current sensors for methane, consuming not more than 16 mA at 8-18 V dc, are required by the Bureau of Mines for its Intrinsically Safe Mine Monitoring System (ISMMS) project. No commercially available methane sensors can operate within the ISMMS power and current limitations. This report examines the feasibility of developing methane sensors that can meet the ISMMS requirements. Of several novel approaches evaluated, the following three are recommended as the most promising: (1) direct electrochemical detection of methane, (2) reconfiguration of present hot-wire sensors, and (3) electrochemical detection using precatalyzed samples.

1 INTRODUCTION

As part of its Intrinsically Safe Mine Monitoring System (ISMMS) project, the Bureau of Mines (BOM) has established stringent criteria for a new type of methane sensor. This sensor must use an extremely low level of current and consume much less power than currently available devices (see Table 1).

The study described here had two objectives. The first was to explore methods of reducing the operating current for existing sensors without sacrificing any of the present benefits, such as long service lifetime, stability between monthly calibrations, rapid response time, and resistance to catalyst poisons or common types of interference. The second objective was to investigate the state of the art of other detection technologies, particularly electrochemical technologies, to identify devices or principles that might be used to develop sensors that meet the BOM criteria.

To accomplish these objectives, information available on commercial instruments (see Appendix) as well as the scientific, technological, and patent literature (see Sec. 2) were surveyed in detail. This survey information was evaluated (see Sec. 3) and used to develop the recommendations presented in Sec. 4. Several brief laboratory investigations also were performed to fill gaps in the literature and to test the feasibility of some of the specific recommendations.

Table 1 Methane Sensors for the Bureau of Mines ISMMS

| Sensor Characteristic | Critical ISMMS Requirement ^a |
|---------------------------------------|--|
| Maximum power | $\begin{array}{l} \text{i} \leq 16 \text{ mA} \\ \text{E} \leq 818 \text{ V dc} \end{array}$ |
| Sensing range | 0-5% methane |
| Accuracy between monthly calibrations | \pm 0.15% at 0.5% methane in air \pm 0.2% at 1-2% methane in air \pm 0.3% at 3-4% methane in air |
| Environmental stability | Can withstand mine environment for at least one year |
| Response time | \leq 60 s to reach 90% signal, continuous |
| Specificity and poisoning | No response to CO, CO ₂ , or H ₂ O vapor Not poisoned by silicones Not affected by outside accuracy specification |
| Output | Proportional to CH ₄ concentration |

^aFrom ISMMS project data, Bureau of Mines.

Certain techniques — such as gas chromatography, mass spectrometry, photon spectroscopies (such as photoionization or infrared), colorimetry, and combination methods (wet chemistry and gas-chromatography/mass-spectrometry) — were excluded from consideration early in the study because the extensive research required to develop them would probably not allow the production of a sensor in the near future. Some methods were excluded because they were not real-time methods or could not meet the power-consumption criteria without an extensive (and expensive) commitment to research and development. The authors' judgments about the ease and cost of development and the probability of success were used to select the final techniques recommended for further consideration.

2 LITERATURE SURVEY

2.1 COMMERCIAL LITERATURE

The Appendix lists companies whose product literature was surveyed. These companies include manufacturers and distributors of gas monitoring instruments used for industrial hygiene, safety, process, and scientific purposes. Some auxiliary equipment manufacturers or distributors are also included.

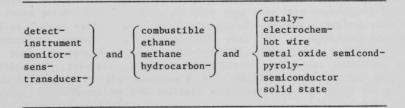
The most promising methane sensors found are manufactured by Scott Aviation and National Mine Service Co., both of whom produce a portable, handheld methane monitor. As far as could be determined from company literature, these monitors meet all of the ISMMS specifications except low power dissipation. The Scott Aviation system required the lowest current: only 60-75 mA.

While resistance to poisoning, smaller size, and improved stability were the stated goals of some manufacturers for product improvement, no indication was found in the literature of any industrial efforts to reduce power consumption.

2.2 SCIENTIFIC AND PATENT LITERATURE

A preliminary search showed that the *Chemical Abstracts* computerized data base was suitable for identification and retrieval of pertinent literature citations; the preliminary search recovered all but the oldest references already in hand. Table 2 shows the key words and combinations of words used to select citations; use of this search profile produced 156 citations for the period January 1970 to August 1982, inclusive. In addition, a hand search of *Chemical Abstracts* was made for 1980 and 1981.

Table 2 Search Profile for Solid-State
Electrochemical Methane Sensors



As expected, fully two-thirds of the available literature consists of patents. Almost all of these involve minor modifications of existing metal-oxide-based sensors rather than development of new technologies. Improved sensitivity to methane, an often-repeated objective, clearly has been a long-standing goal in the industry.

3 APPROACHES TO DEVELOPING A LOW-POWER, LOW-CURRENT SENSOR

3.1 ELECTROCHEMICAL CELLS WITH CATALYSTS

Recent research at Argonne National Laboratory (ANL) has led to the development of a method in which methane is converted to highly electroactive products through the use of heated catalysts. These products can be directed into electrochemical cells with air-permeable electrodes, such as those commonly used to monitor carbon monoxide. This method enables use of the constant-potential amperometry technique to detect a number of organic compounds that otherwise are not electrochemically reactive (such as benzene, hexane, and ethane), and it also suggests means of distinguishing between such compounds.*

The catalyst is normally a noble-metal filament, similar to that used in a hot-wire detector. Use of this type of a catalyst might seem to negate the power-saving advantages of electrochemical detection. However, the filament does not need to operate continuously or even at a temperature high enough to cause complete combustion, particularly when the analyte gas concentrations are high, as is the case with the methane encountered in mines. Conversion efficiencies as low as 0.01% would provide enough partial oxidation products to elicit a response from the highly sensitive electrochemical cells. The high power consumption generally associated with hot-wire detectors can be substantially reduced by making relatively minor changes in geometry (see Sec. 3.3) or by operating the catalyst for brief duty cycles (see Sec. 3.6).

The signal obtained for methane in one of our prototype sensors is shown in Fig. 1. Similar effects have been observed for several other hydrocarbons. This type of combination sensor is still experimental.

3.2 DIRECT ELECTROCHEMICAL OXIDATION

The direct electrochemical oxidation of methane is an approach that could be used to provide a highly sensitive instrument. Normally, the potential required (>1.4 V relative to the reversible H₂ electrode) would preclude the use of aqueous electrolyte sensors, since water may be oxidized at these potentials. Problems of this type have been encountered before and solved through variations of sensing-electrode catalyst, catalyst preparation, electrolyte composition, cell geometry, etc. Use of nonaqueous electrolytes—such as propylene carbonate, glycols, and solid polymers—is a particularly promising approach. Moreover, efficient reactions are not needed because of the relatively high methane levels to be detected in mines. Experiments at ANL indicate a concentration of about 2% methane in

 $[\]star$ This method and the indicated improvement in selectivity are the subjects of ANL patent disclosures.

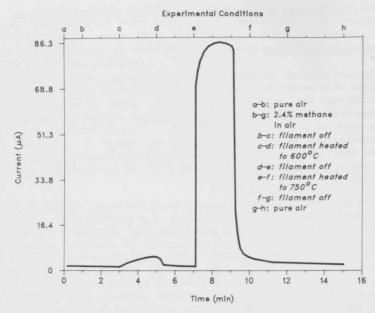


Fig. 1 Typical Responses of a Commercial Amperometric Electrochemical Sensor to 2.4% Methane in Air at 20°C, with and without a Heated Platinum Filament Precatalyst (Samples were passed through a zero filter at a flow rate of 50 cm³/min. Platinum filament diameter is 0.001 in. Sensing electrode is platinum black at 1.1 V vs. a reference hydrogen electrode.)

air can be detected with a platinum-black electrode in a novel sensor. This preliminary experiment is only a first step in the development of a practical sensor; however, the necessary research would not be extensive, and the potential for meeting ISMMS goals is excellent.

3.3 RECONFIGURATION OF PRESENT HOT-WIRE SENSORS

The Scott Aviation transducer mentioned in Sec. 2 draws 60-75 mA at about 6 V dc. Its total power consumption of about 0.4 W is therefore not much greater than the maximum ISMMS specification of 0.288 W (16 mA at 18 V dc).

The Scott Aviation transducer appears to use a palladium oxide catalyst heated by a fine platinum filament (about 0.0005 in. in diameter) of the type disclosed in Ref. 4. If the platinum could be replaced by or coated on a

metal or alloy having a higher electrical resistivity (such as titanium, zirconium, Kovar, or Pt-10% Ni) with the basic structure of the Scott sensor otherwise unaltered, the modified sensor might be made to operate at a current drain of not more than 16 mA at 18 V dc. Alternatively, the filament diameter could be reduced by a factor of 3-4 using novel manufacturing techniques, such as vapor deposition on a suitable substrate and either etching or electroplating to final size.

3.4 LOW-POWER METAL OXIDE CATALYSTS

The existing metal-oxide-catalyst (semiconductor) Taguchi gas sensor is effective only at elevated temperatures because an oxidation (combustion) reaction occurs between the analyte gas and the ionized oxygen that is adsorbed on the semiconductor surface. The depletion of the oxygen ions changes the chemical nature and physical structure of the surface and affects its electrical conductivity. Other semiconductor catalysts have been used and mechanisms explaining their sensing behavior have been proposed. 6,7

Currently available sensors are enclosed in small thimbles of flame-arresting screen. This design provides little resistance to convective loss of heat from the detector element. In principle, a sensor reconfigured to conserve energy would lose only the heat required to bring the sample gas to operating temperature. Because the specific heat of air is low — about 0.0003 $(cal/mL)/^{\circ}C$ — a substantial volume of air can be heated with a few millipoules of energy (Fig. 2). With assumed arbitrary limits for power dissipation, the sampling flow rates are in a reasonable range. For a sensor operated at 500°C, which is a reasonable temperature for methane detection (see Fig. 3), an ideal configuration may sustain a flow rate of 3 mL/min at 35 mW (7 mA at 5 V).

Such a device could be constructed in a tubular form, surrounded by two insulating layers (Fig. 4). In this configuration, most of the heat energy is used to heat the incoming air sample to the catalyst temperature; the chimney design drives the sample through the tubular device by convection. The rate of convection is controlled by selecting appropriate filters. Precise temperature control is managed by the electronics, but the flow resistance of the filter stack sets an upper limit on power consumption. Response time for an internal volume of about 1 mL can be as short as 10 s. In a vertical orientation, convection would carry the sample gas across the catalytic surface, and a stack of filters would limit the flow rate. If flow rates are low and the filter stack is operated upside down, clogging from particulates may be minimized.

Specificity could be achieved if two or more sensors were stacked within the tube, with the upper ones operating at the higher temperatures.

Complementary-metal-oxide-semiconductor (CMOS) circuitry would control the actual input power to the device to control the temperature; high currents

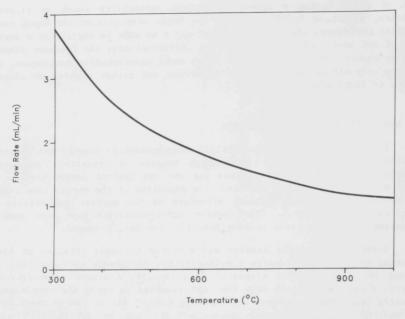


Fig. 2 Flow Rates of Air Necessary to Remove 0.024 W of Power Dissipated as Heat with Incoming Air at $20\,^{\circ}\text{C}$

would only be drawn during the initial heating phase. Similar high-efficiency tube furnaces have been constructed for other uses. However, the effects of the environment (temperature, pressure, and relative humidity) on the performance of this design are not known.

Although it is only hypothetical, this rationale suggests that, with relatively minor modification, Taguchi gas sensors could be made to approach ISMMS specifications. However, other problems encountered with this sensor, such as poisoning, do not appear to be as easy to solve.

3.5 OTHER SOLID-STATE APPROACHES

Some investigators are fabricating palladium—gate hydrogen—sensing devices that dissipate no more than 50-100 mW of heat. Modern thin—film and semiconductor manufacturing technology can be used to produce extremely small devices. Heating elements may be vapor deposited onto silicon or ceramic substrates and coated with catalyst or used directly as a hot—wire type of sensor. Problems could be encountered with this type of sensor in the

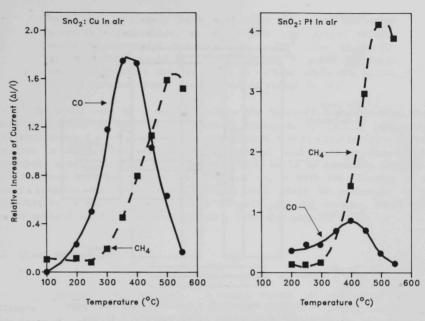


Fig. 3 Response Characteristics of Metal-Oxide-Catalyst (Semiconductor)
Sensors Optimized for Carbon Monoxide (100 ppm)
and Methane (10,000 ppm) (Source: Ref. 7)

following areas: stability of the structures; conductivity of the substrate; longevity of very small, hot films; and cost.

3.6 DUTY-CYCLE APPROACHES

In principle, some sensors that satisfy all of the ISMMS criteria except low power consumption can be brought within the specified limits for power consumption by reducing the duty cycle. The limitations to this approach are as follows:

- The heat capacity of the heated element must be very small in order to bring the element to a constant temperature reliably and accurately and to provide a rapid cooling rate.
- The required power must be stored so that the instantaneous drain on an external power supply is never greater than 16 mA. About 1000 μF of capacitance is needed to store enough

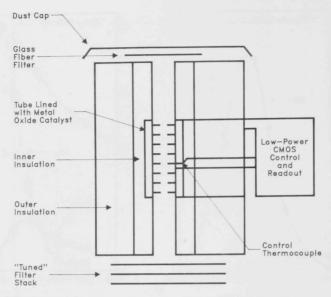


Fig. 4 Configuration of a Hypothetical Low-Power Metal-Oxide-Catalyst (Semiconductor) Sensor

power to sustain a 0.3-s pulse of 60 mA at 18 V. It remains to be shown that pulse durations of about 0.3 s can yield satisfactory accuracy and sensitivity for the measurements. The switching of power into the sensor at turn-on time also must satisfy ISMMS criteria. Use of semiconductor switching with no metal-to-metal contact should permit this requirement to be met. Alternatively, the external power supply might be designed to supply a higher current intermittently to each transducer, without exceeding a maximum total current of 16 μA per transducer. However, it must be ascertained that such an approach could be made compatible with the ISMMS requirements.

• The sensor must either be insensitive to minor shifts or inaccuracies in temperature or be subject to tight control of power dissipation. For example, hot-wire sensors with platinum filaments display the characteristic increase in resistance as they are heated; that property is used in the measurement of the filament temperature. The voltage and current (monitored electronically) can be used to compute the resistance with an analog divider circuit applied to a feedback control. At turn-on time, the initial power drain

would be relatively high until the filament approached operating temperature, at which time the applied voltage would be decreased automatically. This type of servodriven cycling would be very economical of power and would permit very short duty cycles. At no time would the demand on the external power supply need to exceed 10 mA.

The power used by a sensor such as the Scott Aviation hot-wire device could be reduced as much as an order of magnitude by cycling (e.g., 0.3-s operation every 3 s to guarantee a fast response time). Similarly, a hot-wire catalyst in combination with an electrochemical cell could be operated in a duty-cycle mode. For this device, such a mode would be desirable because the cell response with and without the filament on, or with the filament at different temperatures, would yield outputs that could be processed to gain selectivity. A cell current with the filament off, for example, would not be due to methane at the sensing electrode potentials normally used.

3.7 OTHER METHODS

Low-Temperature Catalysts. A small amount of literature describes attempts to develop catalysts that would be effective at low temperatures. For example, a Matsushita Electric Industrial Co. patent describes the preparation of a manganese dioxide catalyst with a reported effective temperature of $70\,^{\circ}\text{C}$ for carbon monoxide and $150\,^{\circ}\text{C}$ for methane. Less dramatic results have been achieved by simple empirical screening of various catalysts and dopants. 13,14

Although it may be possible to develop such catalysts, it is difficult to imagine how they could be operated without serious contamination and interference problems over long periods of time. At $150\,^{\circ}\text{C}$, the catalyst is unlikely to be self-cleaning and its performance would be expected to be susceptible to fluctuations in relative humidity. These characteristics would pose problems for an effective mine-monitoring system.

Biological Catalysts. Many bacteria¹⁵ can consume methane as their sole source of energy. The first step in the metabolism of methane is the addition of an oxygen atom to the substrate. Preliminary evidence shows that the enzyme catalyzing this step contains a porphyrin, similar to that in hemoglobin, but with nickel instead of iron in the central position. 16,17 Suspensions of burst bacteria continue to oxidize methane for a long period of time. If the biocatalyst is that stable in vivo, it might be possible to stabilize it in vitro by affixing it to a polymer matrix. This is a technique common in the industrial use of enzymes. It is equally likely, however, that these enzymes may be unstable, have short lifetimes, and be strongly temperature-dependent. The potential outcome of such an enzyme technique

would be the development of sensors that are highly specific to methane and operate at room temperature. As almost no information is available on the enzyme system, this approach is classified as a basic high-risk, high-payoff approach.

Exclusion Chromatography. A suggestion for using much-simplified gas chromatography to analyze gases, especially methane and other small hydrocarbons, was found in a French paper. In the device described in this paper, a continuous stream of sample air is passed through a short column containing molecular sieve. Instead of an injection of sample, a filament in the air stream is briefly turned on, removing methane and producing carbon dioxide, water, and pyrolysis products. These products are separated in the column by exclusion chromatography and measured with a differential thermal conductivity detector. The resultant patterns of combustion products eluting from the column are characteristic of the gas present in the air. The authors claim high specificity for methane, with only hydrogen interfering.

Adaptation of such a scheme to ISMMS standards would depend on two conditions. First, there would have to be a source of sample air under some pressure (in a ventilation system, for example) because it would be difficult to operate a reliable pump within the 10-mA limit. (Alternatively, a source of vacuum would serve the same purpose, but may involve unacceptable distribution problems.) Second, a low-power sensor, such as an electrochemical cell, would have to serve in place of the thermal-conductivity detector.

This method could potentially be used in a portable monitor, where the power drain of a pump is less critical; it would, however, probably be mechanically complex, involve a substantial data-processing load, and require highly critical components. In addition, the method is inherently discontinuous, but this need not result in long detection times if the cycles are short enough.

4 RECOMMENDATIONS AND CONCLUSIONS

Although no methane transducer is currently available for the ISMMS project, such a device can probably be developed in the near future using one of the approaches described in this report. The recommendations listed below outline suggested experimental and developmental paths to accomplish the BOM objectives in the most timely and cost-effective manner. The recommended approaches are listed in order of priority.

4.1 DIRECT ELECTROCHEMICAL DETECTION OF METHANE

This approach appears to be the most feasible; preliminary ANL experiments suggest that adequate electrochemical signals can be obtained for methane. Although this approach entails some risk, the risk is not very high, because feasibility has been demonstrated. The potential payoff, on the other hand, is very great. Electrochemical cells currently in use for other vapors satisfy most or all of the ISMMS criteria (except methane specificity): low power, low current, minimal zero drift, long life, stability, and resistance to contamination or interference.

The research at ANL (in a cell designed for other vapors) revealed some of the technical difficulties that must be addressed before such a device can be built. Further studies will be needed to determine the optimum combination of electrode catalyst and electrolyte, as well as the most appropriate potential(s) for cell operation.

4.2 RECONFIGURATION OF PRESENT HOT-WIRE SENSORS

Some currently available hot-wire sensors approach the BOM requirements, but none has sufficiently low power consumption. Some companies have developed moderately low-power devices that draw 50-100 mA. A program designed specifically to address the problem of current drain should, therefore, be able to yield sensors drawing not more than 16 mA at 8-18 V dc.

Several approaches have been considered. Present low-power sensors, such as those supplied by Scott Aviation, should be obtained and remounted in test jigs that minimize heat loss and maintain an acceptably high convection-driven sample flow rate. Electronic apparatus to operate the filaments in a 10% duty cycle should be designed; the drain on the external power supply would not exceed 16 mA at any time, although the filament itself may draw more current from an internal capacitive circuit during heating.

A second approach worth considering is to apply novel manufacturing techniques to the Scott Aviation sensor to increase the resistance three— to four—fold or to reduce the filament size or do both. Small filaments may be vapor—deposited on suitable substrates and either etched or electroplated to

final size. Such filaments would have to be very uniform to avoid wire burnout and substrate separation. However, the power dissipation allowed ($\leq 0.2-0.3$ W) would not approach the limits of technology for modern microstructure fabrication.

4.3 COMBINED ELECTROCHEMICAL AND SOLID-STATE SENSORS

A final approach recommended for the development of an instrument with the potential to meet ISMMS criteria is the combination of hot-wire and hot-catalyst pyrolysis with electrochemical detection. Methane in contact with a hot catalyst wire is converted partially to carbon monoxide and formaldehyde, which are further oxidized and detected at the cell electrode. For these purposes, the filaments need not be large, operated continuously, or particularly hot. This method has been developed recently and is not yet in regular use, but its feasibility has been demonstrated in extensive ANL laboratory studies.

Section . The dependent

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APPENDIX:

FIRMS PRODUCING OR DISTRIBUTING SENSORS
OF POSSIBLE INTEREST TO ISMMS

.

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